# Modeling and Control of Advanced CVD Processes by PRS

### FINAL TECHNICAL REPORT

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# **Objectives**

As part of an existing DoD-MURI Center research is being conducted in the field of real time optical process monitoring and control involving a close collaboration between Physicists, Materials Scientists, Chemical Engineers and Mathematicians. In this effort students address specifically closed-loop control of pulsed chemcial beam epitaxy (PCBE) of compound semiconductors utilizing a multiple set of optical sensor inputs. These real time techniques are in development under the existing DoD MURI funding, F49620-95-1-0447, entitled "Modeling and Control of Advanced Chemical Vapor Deposition Processes: the Control of Defects in Mixed III-V Compound Heterostructures," of which Prof. Dietz and Prof. Ito are Co-Investigators. These students have worked with Profs. Dietz and Ito on the acquistion of experimental input data for the real-time modeling of PCBE processes and on the development and implementation of control theory algorithms.

### Status of Efforts

Students have played an integral part in the development and testing of PRS sensing and real time feedback control in pulsed chemical beam epitaxy (PCBE) experiments. Filters and control algorithms have been successfully implemented in PCBE experiments to control growth of thin films of  $GaP/Ga_{1-x}In_xP$  on Si substrate.

# Accomplishments and New Findings

During the duration of the AASERT grant three students were supported. They participated in several phases of the effort.

The students implemented the thickness and compositional controlled epitaxial growth of  $Ga_{1-x}In_xP$  heterostructures on Si(001) substrates, apply-

ing p-polarized reflectance spectroscopy (PRS) as real-time feedback sensing technique. The mathematics and control underlying these efforts was developed with the assistance of graduate students S. Beeler, T. Simon and V. Woods. V. Woods' thesis focused on the real-time thickness and compositional control of  $Ga_{1-x}In_xP$  heterostructures under pulsed chemical beam epitaxy (PCBE) conditions and the ex-situ characterization of the grown epilayers. He closely interacted with the I. Lauko and K. Ito in the implementation of the control algorithms and the establishment of the dataexchange links between process control software/computer and the remotely stationed simulation computer and its software. For closed-loop control, nonlinear control algorithms that utilizes the PR signals to control thickness and composition during heteroepitaxial growth have been developed and tested. An extended optical data base was established from more than one hundred growth runs, which entered in the knowledge base of the control program. The data base links the real-time optical PRS data with ex-situ established film properties, such as composition and thickness.

For ex-situ characterization of the grown strained  $Ga_{1-x}In_xP$  heterostructures by Raman spectroscopy, Mr. Woods went from May 06 till July 04, 1999 to the Technical University Berlin (TUB) and the Hahn-Meitner-Institute (HMI), where he interacted with the research groups of Prof. Hoffmann and Prof. Lewerenz. During this time he was trained in Raman spectroscopy and gained experience in analyzing his Ga1-xInxP layers. This international research exchange was supported by an exchange agreement between the institutions and has been proven an excellent opportunity for students. Back at NCSU he continued to apply this knowledge on the newly build up Raman spectrometer, purchased through a AFOSR/DURIP grant

this year. Vincent Woods completed his thesis on this topic in March 2000, received his degree and joined Microcoating Technology Inc., in April 2000.

We explored the thickness and compositional control of epitaxial  $Ga_{1-x}In_xP$  heterostrucutes on Si(001) substrates, applying p-polarized reflectance spectroscopy (PRS) as a real-time sensing technique. The high surface sensitivity of PRS enables us to move the control point close to the point where the growth occurs, which in a chemical beam epitaxy process is the surface reaction layer (SRL), built up of physisorbed and chemisorbed precursor fragments between the ambient and film interface. The decomposition kinetics during pulsed supply of organometallic precursors has been described by a reduced order surface kinetics (ROSK). The ROSK establishes the links between surface reaction chemistry, composition, deposition rate, film properties, and to the PR response, which is monitored at two wavelengths and two angles of incidence.

Both S. Beeler and T. Simon participated in the development of stack layer models for surface growth, development of ROSK models and development of PRS sensing formulas. Simon completed her Ph.D. in applied math in December, 1999 (now at Intelligent Systems, Inc.). Beeler will defend his Ph.D. thesis in applied mathematics, October, 2000.

#### Personnel Supported

V. Woods, S. Beeler and T. Simon

#### **Publications**

 S. Beeler, H.T. Tran and N. Dietz, "Representation of GaP Formation by a Reduced Order Model Using P-Polarized Reflectance Measurements", J. Appl. Phys., (1999), to appear.

- 2. I. Lauko, K. Ito, V. Woods and N. Dietz, "Filter Design and Control Algorithms Applied to  $Ga_{1-x}In_xP$  Film Growth Control", Applied Surface Science, (2000) submitted.
- V. Narayanan, S. Mahajan, K.J. Bachmann, V. Woods and N. Dietz, "Island Coalescence-Induced Defects in Gallium Phosphide Layers Grown on Silicon: I. Stacking Faults and Twins:, *Phil. Mag A*, (2000), submitted.
- V. Narayanan, S. Mahajan, K.J. Bachmann, V. Woods and N. Dietz "Island Coalescence-Induced Defects in Gallium Phosphide Layers Grown on Silicon: II. Inversion Domain Boundaries in 001 Layers", Phil. Mag A, (2000), submitted.
- 5. V. Woods, K. Ito, I. Lauko and N. Dietz, "Real-time Thickness and Compositional Control of  $Ga_{1-x}In_xP$  Growth Using P-Polarized Reflectance", J. Vac. Sci. Technol. A 18(4)(2000) pp. 1190-1195.
- V. Narayanan, S. Mahajan, N. Sukidi, K. Bachmann, V. Woods and N. Dietz, "Orientation Mediated Self-Assembled Gallium Phosphide Islands Grown on Silicon", *Philosophical Magazine A* 80(3),(2000) pp. 555-572.
- N. Dietz, V. Woods, K. Ito and I. Lauko, "Real-Time Optical Control of Ga<sub>1-x</sub>In<sub>x</sub>P Film Growth by P-Polarized Reflectanc", J. Vac. Sci. Technol. A, 17(4), (1999) pp. 1300-1306.

# Interactions/Transitions

#### Presentations

- 1. V. Woods, "Real-time thickness and compositional control of  $Ga_{1-x}In_xP$  Growth using P-Polarized Reflectance", AVS 46th International Symposium, Seattle, WA, October 25-29, 1999.
- 2. N. Dietz, S. Beeler, H. Tran, and V. Woods, "Real-time Optical Characterization of Surface-Reaction Kinetics During Heteroepitaxial  $Ga_xIn_{1-x}P$  Growth by P-Polarized Reflectance", at the Twelfth American Conference on Crystal Growth and Epitaxy, Vail, CO, August 13-18, 2000.

# New Inventions/Patents

None

#### Honors and Awards

- S. Beeler, NSF Graduate Traineeship
- T. Simon, GAANN Fellowship.